PROGRESS REPORT
MONSANTO INDUSTRIAL CHEMICALS CO.
Research and Development

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PROJECT TITLE

DIELECTRICS - NEXT GENERATION

SUMMARY

New Capacitor Fluids

DC Life Tests on MCS 1475 and MCS 1588 to determine the optimum stabilizer package for these materials in this type of service have been started. Neither fluid gives as good results with 0.5% anthraquinone alone as is shown with 0.3% Unox 221 alone or with 0.3% Unox 221 plus 0.5% anthraquinone. No failures have occurred after 6720 unit hours of service for these two sets of capacitors. After these tests have been run for a longer time, we should be able to tell whether or not anthraquinone is needed with Unox 221. (RHM)

Aroclor 1016 Pink Fluorescence which has caused concern at G.E. and here too appears related to the use of chlorine from Vicksburg Chemical produced as a by-product from their potassium nitrate plant. Laboratory experiments show that Porocel adsorption removes the pink fluorescing impurity. This means that exclusion of the Vicksburg Chemical chlorine and repacking the Porocel towers in the plant as planned should solve this problem. (RHM)

Toxicity Studies - A program has been set up to extend our knowledge of the toxicity of our non-PCB dielectric fluids beyond the acute toxicity, 105 day rat feeding study and fish toxicity studies that have already been made. Vapor



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inhalation toxicity for each component of our mixtures will be obtained. A screening test for carcinogenicity will also be run. Materials involved will be TXS, MIPB and MCS 1659 for the vapor inhalation studies and MCS 1238, 1475, 1588 and 1633 for the carcinogenicity test. Vapor inhalation data are available on Santicizer 711 already and will be available on Santisol 100 from another study already set up. (RHM)

New Capacitor/Transformer Fluids - Fire resistance seems still to be high on the list of desirable properties for new dielectrics. We have made a compilation of biodegradation data and flammability behavior of various chlorinated diphenylmethanes, (in Table I). From these data it is evident that MCS 1560 and MCS 1558 may well be useful non-PCB "Aroclor" substitutes, particularly since the latter (MCS 1558) has a flash point well above that of Aroclor 1016 or Aroclor 1242. Assuming MCS 1558 is biodegradable (these tests are in progress) the key to its becoming an attractive product is largely one of economics.

MCS 1558 requires trichlorobenzylchloride (TCBC) as a starting material. TCBC was manufactured at WGK (Dept. 267) in 1967. At that time its mfg. cost was 11.52¢/lb. of which 4.36¢ was raw materials - principally toluene and chlorine. Obviously a current mfg. cost would be considerably higher but TCBC could clearly be a reasonably priced intermediate for the manufacture of MCS 1558. Unfortunately up to now, our experience with the next step, the benzylation of benzene, has been discouraging. The difficulty is that benzyl chlorides react faster with a monobenzylated benzene than with benzene itself. Thus one obtains inordinately large amounts of di- and poly-benzylated products and these probably aren't biodegradable. In the case of TCBC and benzene in a 1:3 mole ratio typical of Santosol 100, one obtains only a 39% yield of trichlorodiphenylmethane (MCS 1558) with perhaps another 20% of the TCBC going to dibenzylated products and the rest going to high boiling polyproducts. Such poor yields of monobenzylation are, of course, intolerable and we have addressed ourselves to this problem with encouraging results.

As we see it, a reaction technique is required which will afford maximum opportunity for TCBA to attack benzene while at the same time avoiding, or at least minimizing exposure of the monobenzyl product, to additional TCBC. Theoretically this could be accomplished by causing TCBC to react with benzene



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at infinite dilution. As this is not within economics realities, we have therefore designed a system to approach this dilution in the reaction zone while actually using only a normal 3 or 4 molar excess of benzene overall. This is accomplished by adding a TCBC solution in benzene to a fractionating column packed with metallic aluminum at a steady boilup of benzene into the column and reflux system. Enough HCl is generated to keep an active $AlCl_3$ surface on the aluminum column packing to effect benzylation. The mono-benzylated product, being high boiling, travels down the fractionating column (into the boiler) away from the incoming TCBC at the top of the column. It thus suffers much less secondary attack by the TCBC. The initial experiment using this approach in a makeshift apparatus afforded a 77% yield of mono-product (MCS 1558) with an overall ratio of benzene: TCBC of 4:1. There was surprisinglv little attack upon the aluminum column packing by the HCl generated in the benzylation step even though the reaction proceeded briskly in the top 10% of the aluminum bed. This may well be a useful technique for other benzylations of low boiling aromatics . (QET)

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Pilot Plant - Equipment was set up in the pilot plant for producing drum quantities of MCS 1588. Laboratory blends of the S-100 feedstock for the pilot run revealed a high dissipation factor of 0.17 to 0.25. Lab tests indicated an acid treatment of 0.5% (75% $H_2SO_4$/25% $H_2O$) for 1 hr followed by 2% earth treatment on the S-100 could produce a MCS 1588 with an acceptable Df of 0.06-0.07. Acid refinement was carried out on the pilot scale and produced a MCS 1588 with Df of 0.04-0.05. Although this process produced a quality product, much mechanical difficulty was experienced with the equipment. An alternate refinement technique using selective adsorption showed considerable promise in the lab. Equipment is being assembled to test this approach. E.U.C. ordered two drums of MCS 1588 for further testing. Two drums of the pilot plant material were used to fill their order. P. R. Mollary have requested a meeting the week of March 17 to discuss MCS 1588 vs. MCS 1475.

The Krummrich plant is planning to produce a large batch of MCS 1489 in the plant. Because of bad equipment they are forced to use batch Fullers earth refinement instead of Porocel columns. Since this has not been checked, we plan some tests to anticipate what can be expected in the plant. (JGB)

Dielectrics - Europe - We are determining biomaqnification factors in fish for Aroclors. Measurements of PCB, by solvent extraction from repeat samples of water from one of the Ruabon fish-ponds, have given higher values than those from the polyurethane-foam extraction of samples in October. The reasons for these discrepancies are not understood, but to establish maximum confidence in the results on which to base estimates of biomagnification factors, we will sample all the ponds again and use solvent extraction to determine the PCB levels. (HAV)

Dielectrics

Aroclors - The discontinuing of air-blowing of crude Aroclor in the Aroclor Department has led to corrosion problems from residual HCl. We determined HCl solubility in Aroclor at 250°C and concluded that an increase in temperature would not significantly affect the residual HCl. It has been proposed that a vacuum be drawn on the blow tank and that residual HCl be reduced in this way. This will be checked out in the laboratory. (DRC)



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